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IMPURITIES AND INTERFACE STATES IN THE SiO₂/Si SYSTEM

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Semi-Annual Report

"Impurities and Interface States in the ${\rm SiC}_2/{\rm Si~System}$ "

National Aeronautics and Space Administration Research Grant NGR 36-003-067

Principal Investigator: Alan B. Kuper

October 15, 1966

Abstract

The purpose of this research is: (1) to understand factors contributing to instability in surface properties of silicon planar devices and (2) to provide information which silicon device engineers can use to improve reliability.

Sodium migration in the oxide is being studied because of its importance as a failure mechanism. Combined MOS electrical/radio-chemical analysis is the principal experimental method. This gives direct measurement of impurity distribution that underlies electrical instability data. Research includes:

- 1. Impurity diffusion and drift
- 2. $P_2O_5 \cdot SiO_2$ glass stabilization
- 3. Impurity interaction
- 4. Theory of impurity distribution

Results are in the form of impurity distribution profiles within SiO_2 compared with electrical Si surface data on the same samples. Results of importance in device processing are:

- 1. Na is evaporated from within SiO₂ at above 800°C in dry nitrogen.
- 2. Closely-stacked wafers evaporate Na at an appreciably slower rate.
- 3. $P_{2}O_{5}$ gettering is partly an interface effect.

Results of importance in evaluation and testing are:

- Na may remain near the Si interface in hot-bias tests, even though the SiO₂ appears clean electrically.
- 2. Anion may partly cancel Na after high temperature processing.

"Impurities and Interface States in the SiO2/Si System"

Purpose: (1) To understand factors contributing to instability in surface properties of silicon planar devices and (2) to provide information which silicon device engineers can use to improve reliability.

Introduction

In order to improve reliability and circumvent certain failure mechanisms, the engineer doing silicon planar circuit design, process, and testing needs to know the facts concerning each mechanism.

Within the last two years it has become clear that impurities, principally Na, are responsible for gross surface instability in SiO₂/Si planar circuits. Na was shown electrically (1) to be capable of causing observed instability and later was shown to be present as a residual impurity in the oxide. (2,3) We then went the next step (2) and combined the electrical and radiochemical impurity analysis. This showed directly the correlation between (1) distribution of Na in the oxide and (2) silicon surface potential shifts.

At Case Institute we are continuing to study the effect of impurities under conditions typical of actual process heat treatments, such as diffusion temperatures and times; and under conditions typical of actual device operation and accelerated aging, in particular, "hot-bias" conditions. The present research program includes:

- (1) Impurity diffusion and drift
- (2) P₂O₅ · SiO₂ glass stabilization
- (3) Impurity interaction
- (4) Theory of impurity distribution.

Theory of Combined Electrical/Radiochemical (ER) Method

For a conventional MOS (metal-exide-silicon) capacitor, the flatband voltage (V_{fb}) at which the field in the Si is zero may be computed, given exide thickness and Si impurity concentration. Changes in V_{fb} for nearly-parallel shifts of MOS capacitance-voltage curve inflections are used to obtain changes in negative charge concentration ($-\Delta N_s$) induced in the Si surface. (4) ΔN_s is obtained from

$$\Delta N_s = C_{ox} \Delta V_{fb} / q \tag{1}$$

where

q = | electronic charge

 $\Delta V_{fb} = initial V_{fb} - final V_{fb}$

 $C_{ox} = KA/d$

K = oxide dielectric constant

A = area of the field-plate

d = oxide thickness.

$$\Delta N_s / A \Delta V_{fb} = K/qd = 4.0 \times 10^{10} cm^{-2}$$
 (2)

for d = 5400 A.

Also ΔN_s equals the change in the weighted integral of charge concentration (N(x)) in the oxide, (4)

$$\Delta N_s = \Delta (1/d \int_0^d xN(x) dx).$$
 (3)

Net impurity concentration measured by sectioning and radiochemical concentration profiling is used to compute a similar weighted average (ΔN).

$$\Delta N = \sum_{i} (x_{i}/d) N (x_{i}) \Delta x_{i}$$
 (4)

where $x_i = x$ coordinate of the center of the $i^{\frac{th}{t}}$ section of width Δx_i

 $N(x_i)$ = Net cation impurity concentration change (e.g. Na minus Br concentration) in the section.

Then $\Delta N_s/\Delta N$ = fraction of net charged oxide impurities corresponding to a given ΔV_{fb} .

Research

(1) Impurity diffusion and drift (C. J. Slabinski)

In these experiments, deliberate contamination with radioactive Na ²⁴Br ⁸² is being used in the ER method to obtain profiles of both anion and cation by gamma spectrometry. Four main results have been obtained.

First result is that Na evaporates from the oxide in high temperature heat treatment as shown in Fig. 1 where Na concentration drops at least an order of magnitude in 10 min. at 800°C in dry nitrogen. This is confirmed electrically as shown in Fig. 2. It proved possible to retain appreciable Na within oxide by covering the flat sample with a Si flat as is shown in Figs. 1 and 2.

Second result is that, in high temperature diffusion of NaBr, Br diffuses slower and appears partly to cancel Na⁺. This is shown electrically in Fig. 3 and verified radiochemically at 800°C as shown in Fig. 4.

Third result is that Na dominates NaBr drift results in 200°C

"hot-bias" testing. This is shown in Fig. 5 and 6. Br is evidently not sufficiently mobile at 200°C.

Fourth result is that Na alone does not control Si surface potential in the drift experiments. This is shown in Fig. 7. This is of importance in the interpretation of accelerated aging cycles because the electrically "clean" oxide we see in Fig. 6 and 7 still has appreciable Na near the Si.

More detailed discussion of these results and the analysis will be published in the Proc. of the Fifth Annual Symposium on Physics of Failure in Electronics, preprints of which have been submitted to NASA.

(2) P2O5 · SiO2 glass stabilization (W. Smith)

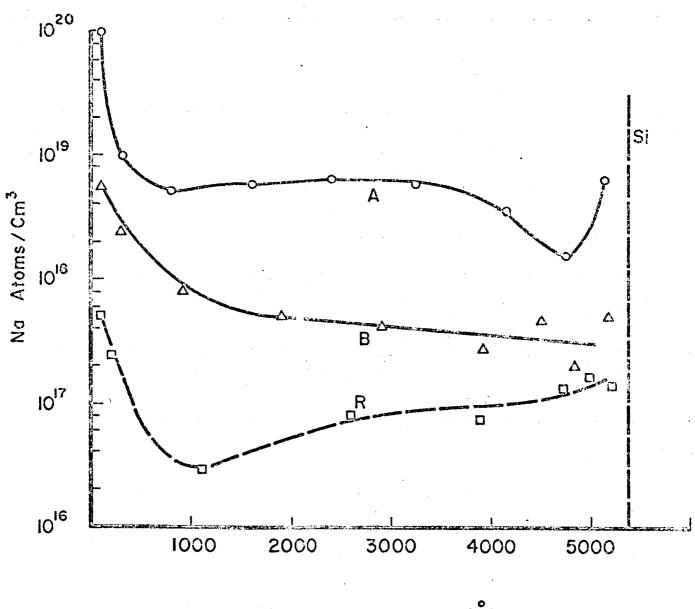
It is well-known⁽⁵⁾ that $P_2O_5 \cdot SiO_2$ glass layers over SiO_2/Si prevent gross surface instability. We showed⁽²⁾ that the glass acted as a getter for Na and in this way explained stabilization.

To understand the gettering mechanism itself we have been fine-sectioning the glass using very dilute HF: water (1:100), measuring etch-rate by interferometry. By etching, it is possible to locate the glass/SiO₂ interface. ER experiments with radioactive NaBr involving both drift and diffusion have been done.

Three main results have been obtained.

(1) Na and Br segregate preferentially not only in the glass but even more in the glass/SiO₂ interface (Fig. 8).

Sodium concentration profile in oxide after 10 minutes at 800 °C by radiochemical analysis of Na²⁴ from NaBr. A, covered sample; B, uncovered. R, typical residual Na profile determined by neutron activation.



Distance Into Oxide (Å)

Fig. 1

Change in room temperature MOS flat band voltage vs. time at 800° C. A, uncontaminated sample, open surface; B, NaBr contaminated, open; C, NaBr contaminated, covered during diffusion.

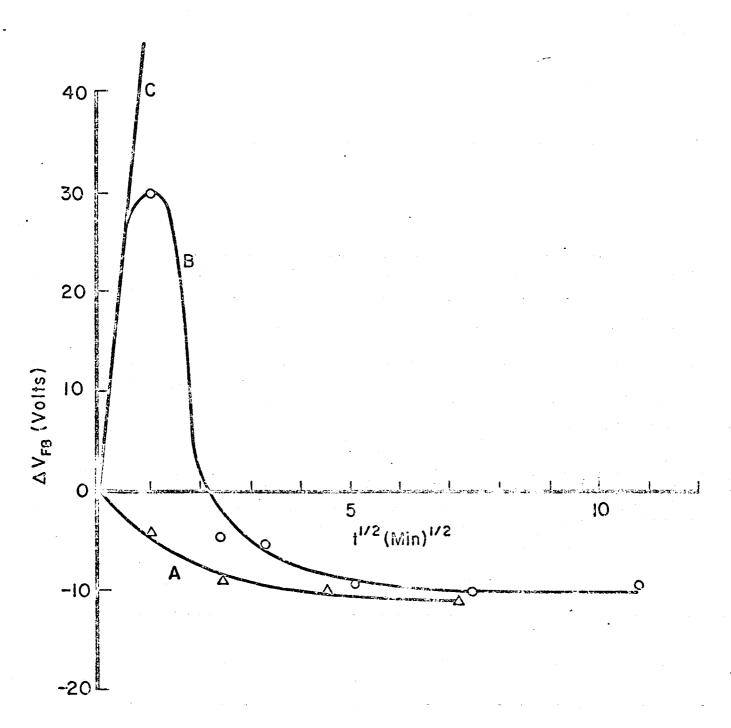
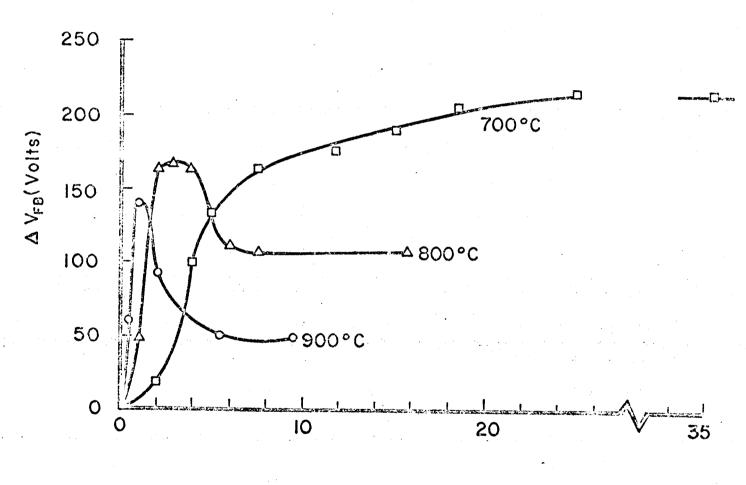


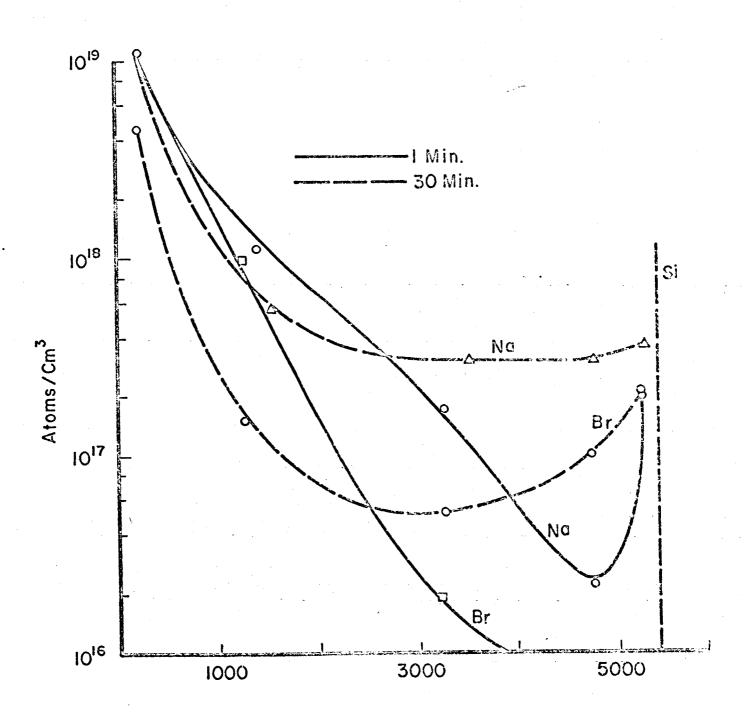
Fig. 2

Change in room temperature MOS flat band voltage vs. time 1/2 at temperature for NaBr contaminated covered samples.



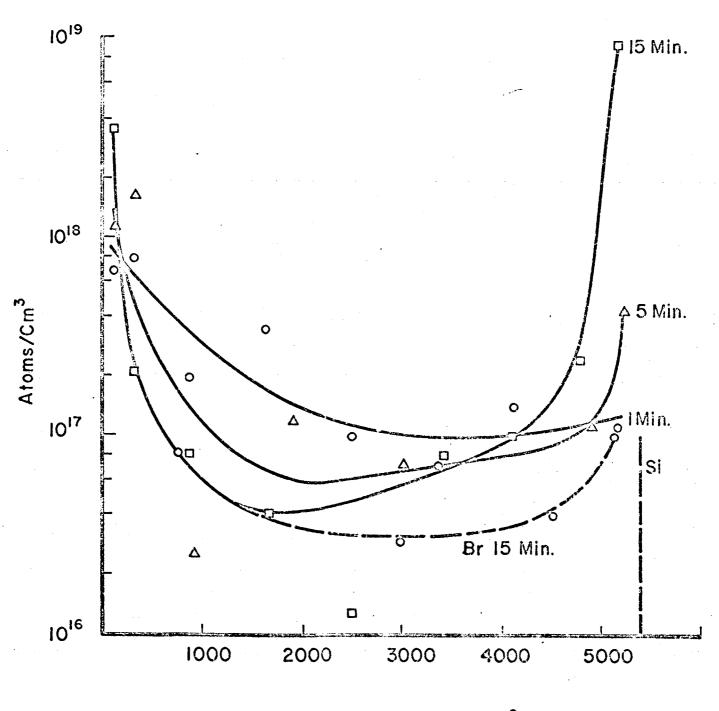
t^{1/2} (Min.)^{1/2}

Na and Br in oxide after 800° C diffusion as in Figure 3. In units of 10^{12} cm⁻², after 1 min: $\Delta N = 6.2$, $\Delta N_s = 4.6$. After 30 min: $\Delta N = 1.7$ cm⁻², $\Delta N_s = 0.8$.



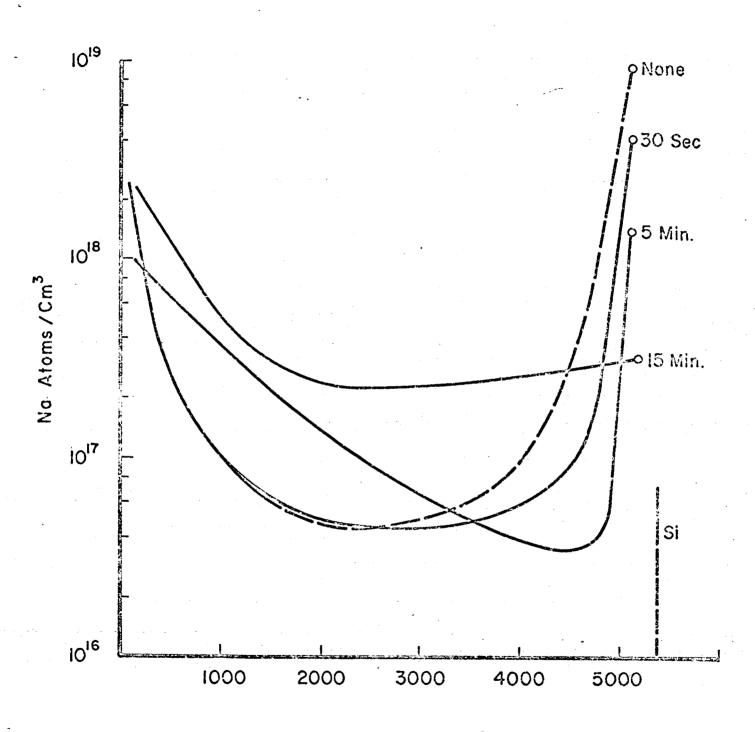
Distance Into Oxide (Å)

Na and Br in contaminated oxide after 200°C forward drift at E = 5×10^{5} volt/cm (metal positive). In units of 10^{12} cm⁻², after 1 min: $\Delta N = 0.15$, $\Delta N = 0.16$. After 5 min: $\Delta N = 17$., $\Delta N_s = 6.4$. After 15 min: $\Delta N = 50$, $\Delta N_s = 9.2$.



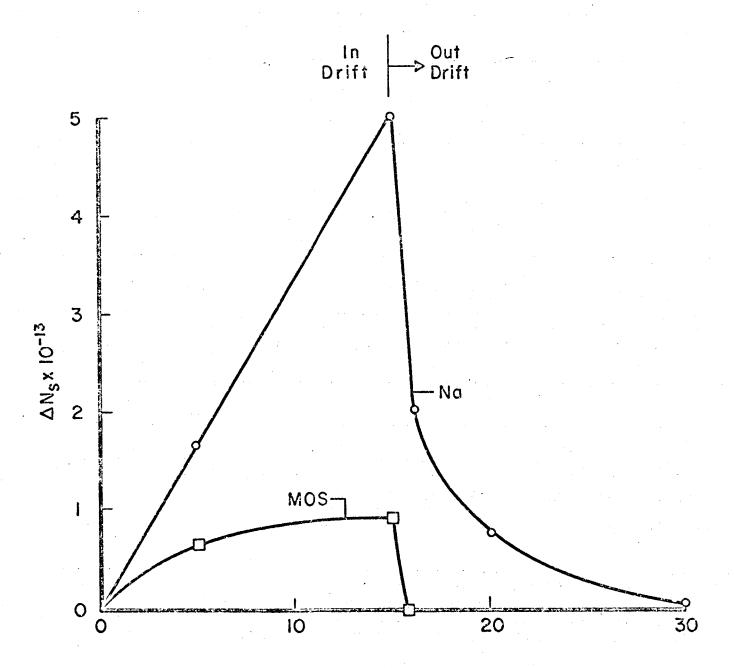
Distance Into Oxide (Å)

Na in contaminated oxide. Reverse drift after 15 min. forward drift. $E = 5 \times 10^5 \, \text{v/cm}$. $T = 200^{\circ} \, \text{C}$. Br profile, not shown, is like that in Figure 5 and is not significant. In units of $10^{12} \, \text{cm}^{-2}$, at start of reverse drift (the sample is that of Figure 5); $\Delta N = 50$, $\Delta N = 9.2$. After 0.5 min: $\Delta N = 20$, $\Delta N = 0.36$. After 5 min: $\Delta N = 7.5$, $\Delta N < 0.04$. After 15 min: $\Delta N = 0.75$, $\Delta N < 0.04$.



Distance Into Oxide (Å)

Comparison of change of charge induced in silicon surface for drift time at 200 °C. Na curve is ΔN computed by equation (4) from radiochemical data. MOS curve is ΔN computed by equation (2) from ΔV_{fb} electrical data.



Time (Minutes)

- (2) This interface spike advances as the glass penetrates the oxide during heating (Fig. 9).
- (3) Sodium can be drifted slightly during hot-bias testing; that is, the glass is not an ideal getter (Fig. 10). This result should enable us to separate Na effects from reported polarization (6) of the glass, both of which shift Si surface potential.

(3) Impurity interaction in SiO₂/Si (G. Holmberg)

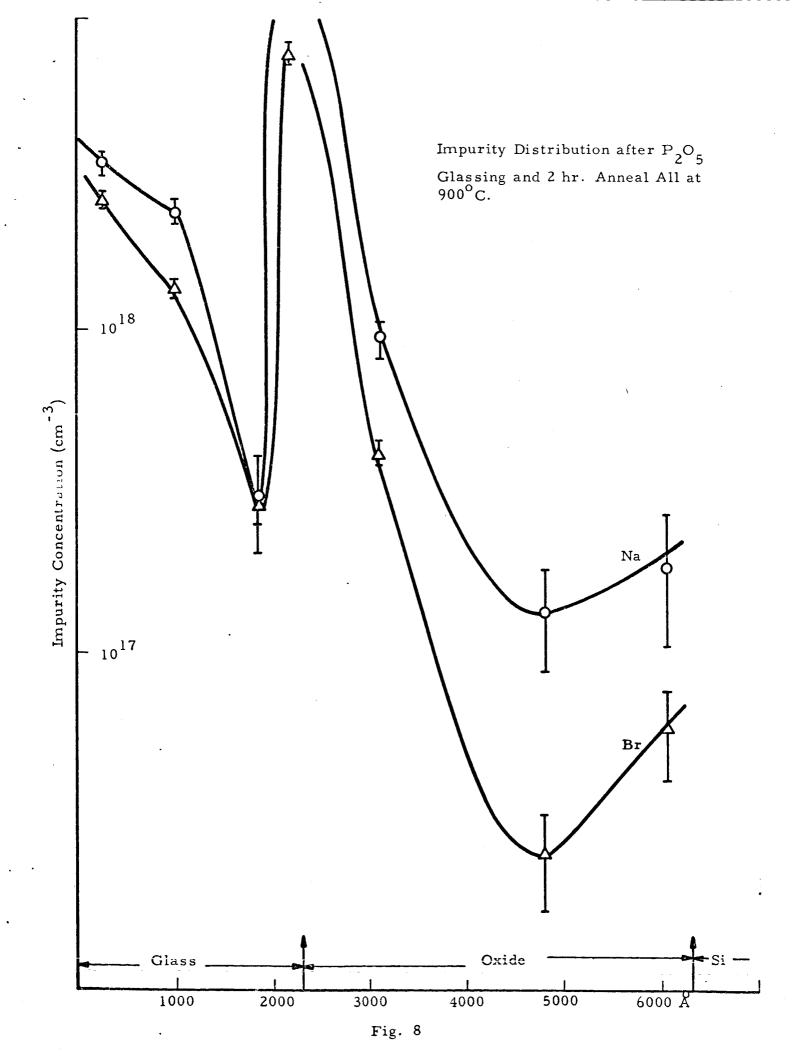
It is known that not only alkali ions but also water and hydrogen affect Si surface stability in the SiO₂/Si system. Since these impurities commonly enter in device process and environment, we investigate possible interrelation between these impurities in SiO₂. For example the field around Na[†] would depend on the presence near it of OH⁻.

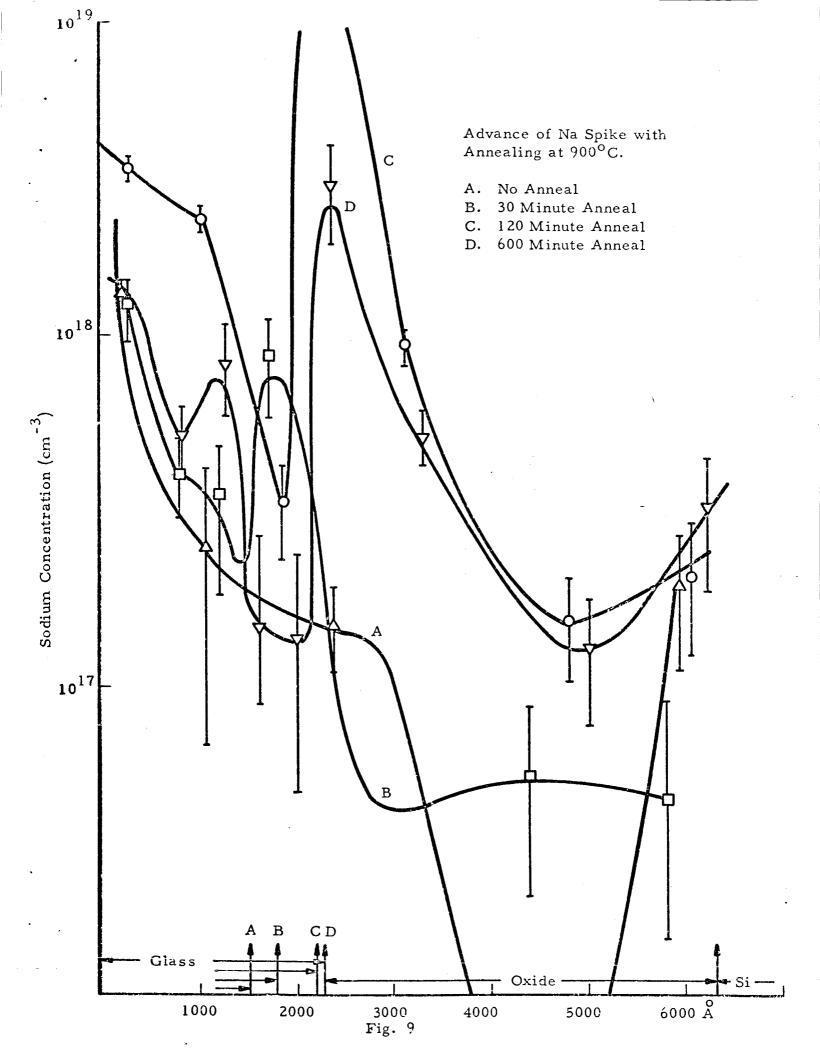
Three main results have been obtained:

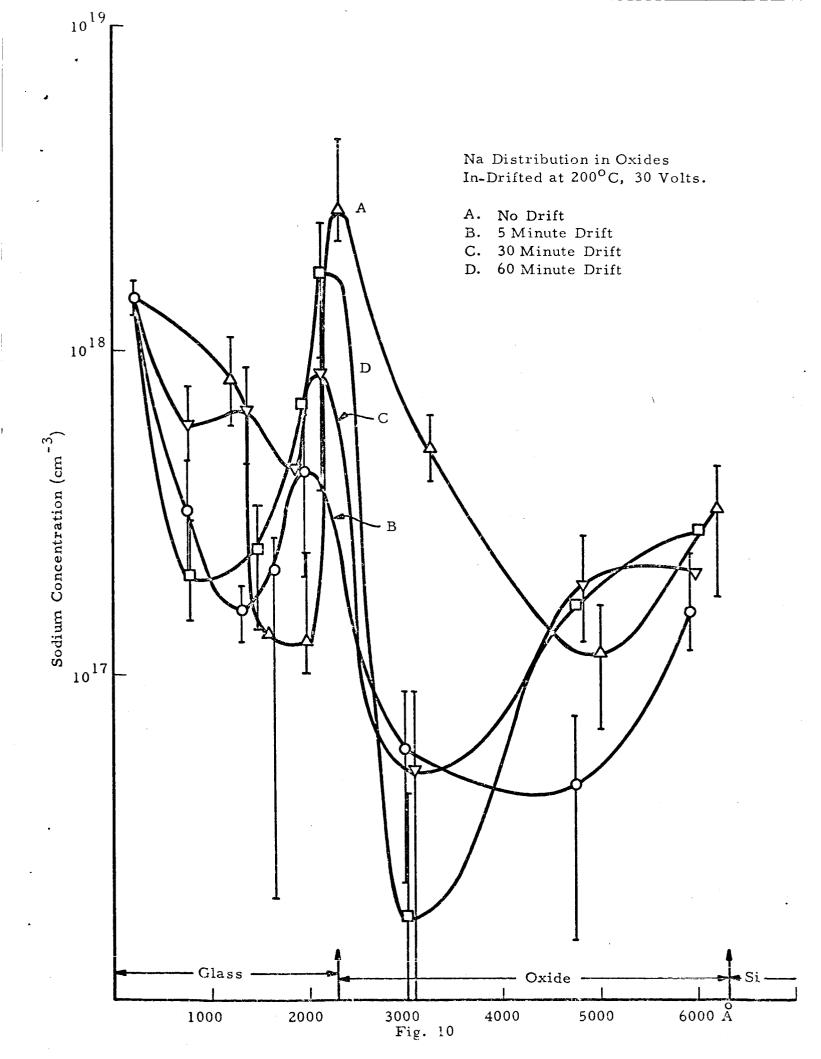
- (1) A survey of pertinent literature.
- (2) A first-order model for water species in the oxide.
- (3) Design and construction of a cell which will permit measurement of MOS C(V) under conditions of controlled temperature and ambient gas pressure. (Fig. 11)

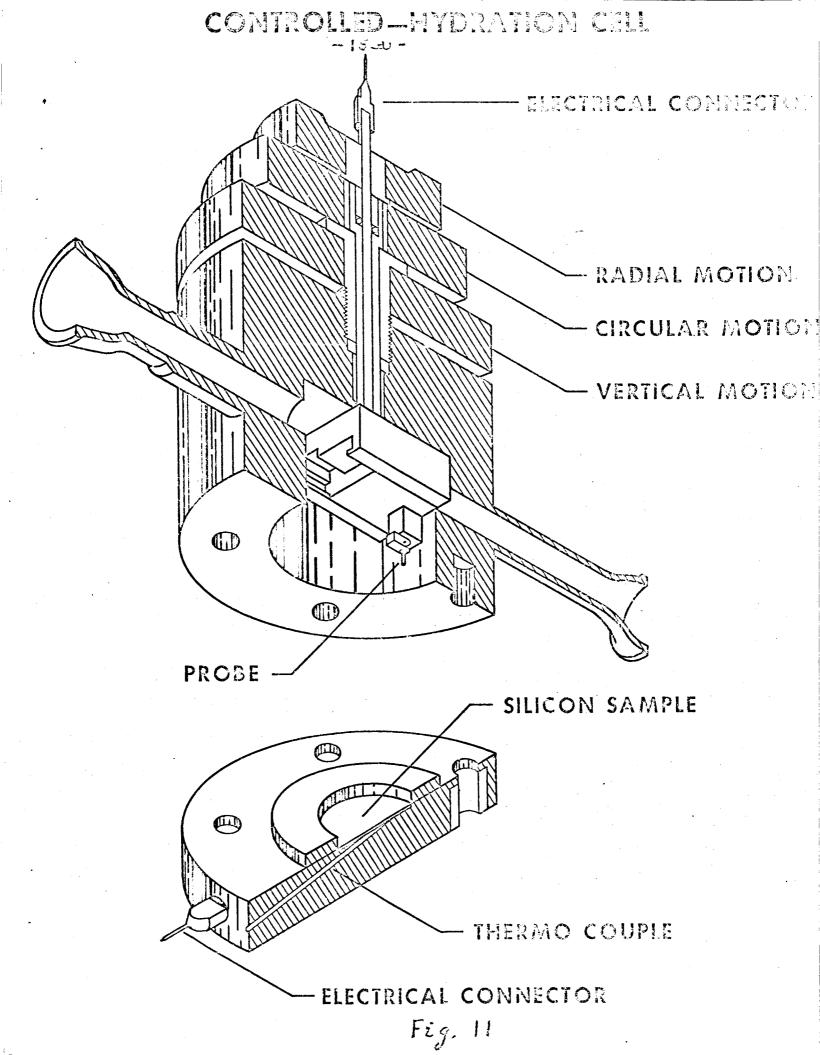
Model for Water in Silica

- (1) Water exists in the silica as hydroxyl, i.e. Si-OH groups, at least at high temperatures. This is indicated by:
 - (a) IR absorption studies. The 2.7 micron absorption in silica is present only when water is present in the silica. It is agreed to be the stretching frequency of the Si-OH bond.









- (b) The half power dependence of solubility of water on the water vapor pressure indicates that the water breaks up into two species upon entering the glass.
- (c) Absorbed water changes all the properties of the silica-viscosity, temperature coefficient of expansion, density--in
 such a way as to indicate looser three-dimensional bonding in
 the lattice than in the absence of water.
- (2) The solubility of water decreases with temperature, but the diffusion coefficient increases with the temperature in the usual way. The diffusion coefficient is concentration dependent, and the concentration dependence is dependent on the diffusion temperature.
- (3) Diffusion of either water or hydrogen into silica produces hydroxyl groups in the silica, as evidenced by the IR absorption produced. If the sample is heated after the diffusion of either hydrogen or water, both hydrogen and water will be evolved. Since both hydrogen and water produce the same chemical effects in the silica, it is quite possible that they will produce the same electrical effects.
- (4) Two types of hydroxyl seem to exist in the glass, one more mobile than the other. Both, however, exhibit the same IR absorption properties.
- (5) Water molecules do not diffuse as such in glass. This is indicated by the fact that an oxygen tracer in a water molecule diffuses a much shorter distance than the hydrogen of the water in the same diffusion time. A proposed model is the diffusion of associated hydrogenhydroxyl pairs.

- (6) In a pure oxide, protons do not move in an electric field, as indicated by drift measurements on oxides grown in tritiated steam. Protons do drift, however, if silica is doped with aluminum.
 Possibly other metal ions will have the same effect as aluminum.
- (7) Water does cause hot-bias instability in electronic devices. Since the instability seems to be caused by positive ion motion, two mechanisms are possible:
 - (a) Protons react with the silica in such a way as to release metal ions, especially sodium, and allow the metal ions to drift.
 - (b) The presence of metal impurities allows the protons of the water to drift.
- (8) Diffusion and solubility of water in a glass are strongly dependent on the thermal processing of the glass prior to the diffusion. Possibly the diffusion of other ions is similarly affected.
- (4) Theory of impurity distribution (H. Caban)

The theoretical treatment of the concentration distribution of Na in a thermally grown film of SiO₂ depends on the particular model under consideration. Models considered so far are different combinations of the following considerations.

Within the oxide, Na moves by diffusion, and, if ionized, by drift under the action of self-generated and applied fields.

The Na may enter the oxide by any or a combination of the following processes:

(1) Rate limited diffusion at the SiO₂/air interface.

- (2) Associated with a negative ion (after which there will be generation (dissociation) and recombination).
- (3) Dissociated (ionized) but accompanied by a negative ion.

 Theoretical analyses have been done of the following:
- (1) Drift and diffusion of fully ionized positively-charged impurity in a metal-oxide-metal (equivalent to accumulated MOS) short circuited, floating, and with applied bias.
- (2) Generation, recombination, drift and diffusion of ions of Na and negative ions (assumed immobile) through a film of oxide with no applied field. As a first approximation, the distribution of the negative ion being assumed as a step function near one face of the oxide.
- (3) Different total amounts of ions of Na and a negative ion (assumed dissociated) moving under diffusion and drift (self and applied electric fields) in a metal-oxide-semiconductor system under bias.

Model 1 is included in this report. Results are given in Fig. 12 for calculated distributions of, for example, Na⁺, across oxide.

We concern ourselves in Model 1 with the steady-state distribution of a positively charged species moving by diffusion and drift under the influence of self and applied fields for a symmetric system consisting of two metal plates separated by a film of homogeneous oxide of thickness x. Across this MOM a constant difference in electric potential is maintained

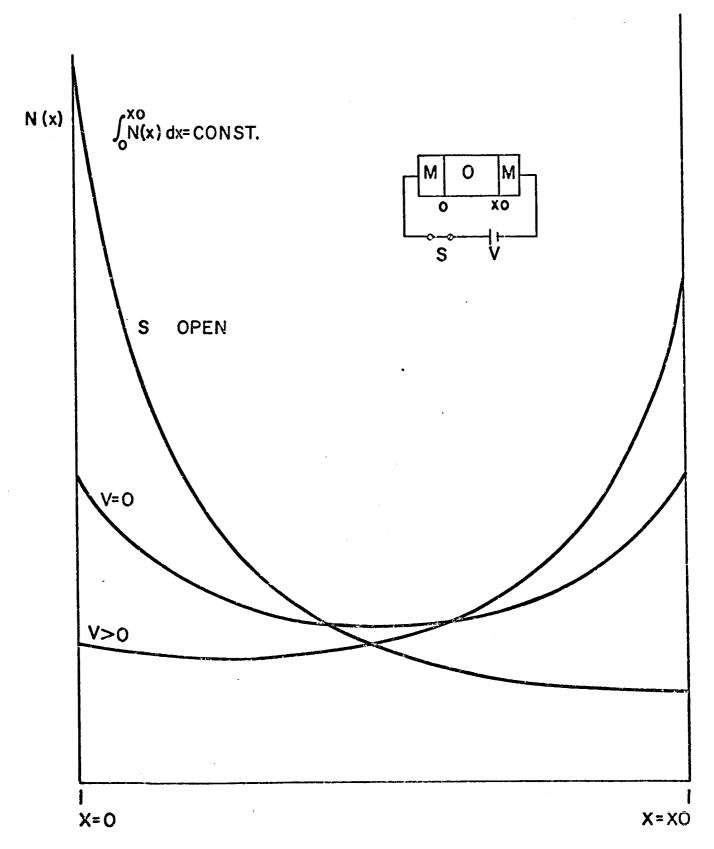


FIG. 12

through an external circuit. We assume that the system at all times retains over-all charge neutrality. This problem is similar to Na⁺ in oxide in an MOS with silicon surface accumulated.

Here and elsewhere, since the actual oxide films under consideration are very thin (less than 10⁴ Å) and have an area around 1 cm. ², we will neglect end-effects, effectively assuming infinite cross-sectional area. The problem, then, is a one-dimensional one and will be solved on a perunit area basis.

For the steady state solution we set the flux of charged particles at any point equal to zero:

$$0 = -D \frac{dN}{dx} + \mu EN$$
 (1)

where N = concentration of charged species

D = diffusion coefficient of the charged species

 μ = mobility of the charged species

E = electric field

x = distance into the oxide measured from the left interface of the oxide.

Solving for E and making use of Einstein's Relation we have:

$$E = \frac{kT}{q} \frac{1}{N} \frac{dN}{dx}$$
 (2)

where Einstein's Relation is given by

$$\frac{D}{\mu} = \frac{kT}{q} \tag{3}$$

We also have

$$\frac{dE}{dx} = \frac{\rho}{\epsilon} = \frac{q}{\epsilon} N \tag{4}$$

where q is the charge of the charged particles and ϵ is the dielectric constant of the oxide. Differentiating (1) with respect to x and using (2), (3), and (4)

$$\frac{d^{2}N}{dx^{2}} - \frac{1}{N} \left(\frac{dN}{dx}\right)^{2} - \frac{q^{2}}{kT\epsilon} N^{2} = 0$$
 (5)

Following the usual procedure for solution, we set

$$P = \frac{dN}{dx}$$
 (6)

upon which (5) becomes

$$P \frac{dP}{dN} - \frac{P^2}{N} = \frac{q^2}{kT\epsilon} N^2 \tag{7}$$

Upon recognizing that

$$P \frac{dP}{dN} - \frac{P^2}{N} = \frac{N^2}{2} \frac{d}{dN} \left(\frac{P^2}{N^2}\right)$$
 (8)

(7) becomes

$$d\left(\frac{P^2}{N^2}\right) = \frac{2q^2}{kT_{\epsilon}} dN$$
 (9)

Due to the movement of the charged species within the oxide film, induced currents will flow through the external circuit and will create an image charge at each metal plate. Since the magnitude of either image

charge is smaller than the total charge within the oxide, it is evident from an application of Gauss's Law that at a certain distance, x m', within the oxide, there will be zero field and therefore from (1)

$$P(x_m) = P_m = 0$$
 $E(x_m) = 0$ (10)

Letting $N(x_m) = N_m$ and integrating (9) from x_m to x we have

$$\frac{dN}{dx} = \pm N \sqrt{\frac{2q^2}{kT_{\epsilon}}} (N - N_{rn})$$
 (11)

where it is evident that the minus sign corresponds to $0 \le x \le x$ and the plus sign corresponds to $x \le x \le x$. Integrating (11) now, we have

$$N = N_{\text{m}} \sec^{2} \left[+ \sqrt{\frac{q^{2}N_{\text{m}}}{2kT_{\epsilon}}} (x-x_{\text{m}}) \right] \qquad (-) \text{ for } 0 \le x \le x_{\text{m}}$$

$$(+) \text{ for } x_{\text{m}} \le x \le x_{\text{c}}$$

$$(12)$$

or translating our axes to $x = x_m$ by letting $x' = x - x_m$,

$$N = N_{\rm m} \sec^2 \sqrt{\frac{q^2 N_{\rm m}}{2kT_{\rm f}}} x' \qquad (13)$$

We may now determine x as follows: from (2) we have

$$d\phi = -Edx = -\frac{kT}{q} \frac{dN}{N}$$
 (14)

And integrating from x = 0 to $x = x_0$

$$V = \frac{kT}{q} \ln \frac{N(x_0)}{N(0)}$$
 (15)

where V is the potential of the left metal plate relative to the right metal plate. Substituting the expressions for $N(x_0)$ and N(0) obtained from (12)

into (14) we obtain, after some algebra,

$$x_{m} = \sqrt{\frac{2kT_{\epsilon}}{2}}_{q}^{2} tan^{-1} \left[e^{-\frac{qV}{2kT}} csc \sqrt{\frac{q^{2}N_{m}}{2kT_{\epsilon}}} x_{o} - cot \sqrt{\frac{q^{2}N_{m}}{2kT_{\epsilon}}} x_{o} \right]$$
(16)

From equations (16) and (13) we see that one effect of the externally applied bias is to translate the distribution curve N vs. x along the x-axis in the direction of the more negative metal plate. If we now require that the total amount of the charged species within the oxide be fixed, then, as the curve is displaced horizontally, N must decrease in order to keep the area under the curve constant.

We now proceed to determine this vertical shift of the curve by finding N_{m} as a function of the applied bias for a given total amount of the diffusing species within the oxide film, N_{T} . From (13)

$$N_{T} = \int_{0}^{x_{0}} N(x) dx = \int_{-x_{m}}^{x_{0}-x_{m}} N_{m} \sec^{2} \sqrt{\frac{q^{2}N_{m}}{2kT_{\epsilon}}} x' dx'$$
(17)

which, after some manipulation, yields

$$N_{T} = \frac{4kT_{\epsilon}}{2} \alpha \left[\cosh \left(\frac{qV}{2kT} \right) \csc \alpha - \cot \alpha \right]$$

$$\alpha = \sqrt{\frac{q^{2}N_{m}}{2kT_{\epsilon}}} x_{o}$$
(18)

where
$$a \equiv \sqrt{\frac{q^2 N_m}{2kT_{\epsilon}}} x_0$$
 (19)

Equations (18) and (19) determine the vertical displacement of the minimum of the N vs. x curve.

The solution to the problem, therefore, is given by equation (12) or

(13) together with equations (16), (18), and (19).

It is interesting to note that from our requirement that N remain finite and equation (13) it follows that $\alpha < \pi/2$ and thus

$$N_{m} < \frac{\pi^{2} kT_{\epsilon}}{2q_{x_{0}}^{2}}$$
 (20)

Finally, for the special case of V = 0 (still retaining the external circuit, however) we have from equation (16)

$$x_{m} = \frac{x_{0}}{2} \tag{21}$$

(which was to be expected from symmetry considerations) and from (18)

$$N_{T} = \frac{4kT_{\epsilon}}{2} \quad \alpha \tan \frac{\alpha}{2}$$
 (22)

A schematic representation of N vs. x is shown in Figure 12.

We may now extend the above solution to the case where the external circuit is removed. Again we assume the system retains over-all charge neutrality. This may be accomplished by depositing the charged species accompanied by an oppositely charged non-diffusing species such that no effective charge is added to the system. We assume that the species are deposited on the left metal-oxide interface where the non-diffusing species will remain.

It is evident that this problem is identical to the previous one, the only difference being in the boundary conditions, which are particularly simple in this case. Since we have assumed over-all charge neutrality,

the field at the right metal-oxide interface vanishes and we have

$$x_{m} = x_{0} \tag{23}$$

and
$$N_{m} = N(x_{o})$$
 (24)

Thus
$$N_{m} = N(x_{o})$$

$$N = N(x_{o}) \sec^{2} \sqrt{\frac{q^{2}N(x_{o})}{2kT\epsilon}} (x_{o} - x)$$
(24)

This case is included with the previous two in Figure 12 where N(x) is shown for V = 0, V > 0, and V floating. Total charge is the same for all three cases.

Discussion of Main Results

Results of impurity diffusion in the present research should be of value in determining optimum process for reliability. For example, making use of our results, a fabrication process which includes a final Na evaporation step with unstacked wafers should make planar circuits less likely to fail due to ion migration. (This assumes, of course, that Na is not introduced in subsequent process.)

Results of our diffusion and drift experiments should be of value in interpreting device failure anomalies. For example, in cyclic hot-bias accelerated aging, the knowledge of Na distribution in oxide which does not appear electrically, can be an aid in interpreting recoverable failure test data.

The result of Na segregation at the $P_2O_5 \cdot SiO_2$ interface in our glass studies may be of value in process for reliability. For example, when removing the glass, it may be important to remove a small amount of oxide as well.

Future Research

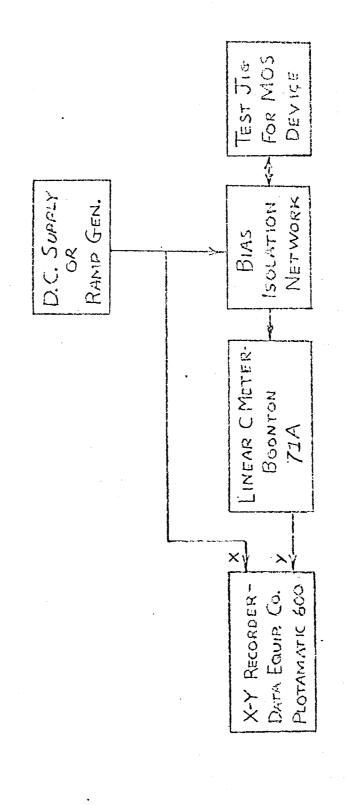
During the remainder of the period of the present grant the research reviewed in this report will continue with the following added scope:

- (1) Diffusion and drift studies will be extended to dry oxides. The kinetics of drift in dry oxide will be studied for differences as compared to wet oxide which should result from the relative absence of OH anion.
- (2) P₂O₅ · SiO₂ studies will be extended to include measurement of Phosphorus distribution, to correlate glass composition with Na impurity "trapping."
- (3) Impurity interaction experiments will be begun with measurement of effect of hydration versus temperature and pressure.
- (4) Theoretical analysis will be extended to more complex cases and boundary conditions. Results will be compared to the experiments. Time-dependent solutions will be considered.

Experimental Equipment and Techniques

Principal improvements in experimental technique have been:

(1) Continuous MOS C(V) plotting system. (Fig. 13) This system makes possible faster and more detailed electrical data-taking than is possible with capacitance bridges nulled at discrete



BLOCK DIAGRAM OF C-V FLOTTING SYSTEM

points. It is especially useful for observing transient changes.

- (2) Faster cooling hot-bias jig.A larger bore Freon line was installed to enable drift samples
 - to be fast-cooled under bias at the conclusion of drift.
- (3) Improved quartz sample container for neutron activation.

 This container satisfies the reactor water-cooled hole size requirements and is easy to unload with long tongs.
- (4) Computer program for radiochemical data reduction.

A computer program has been written to calculate sodium and bromine concentrations in sections of the oxide from etching and counting data. The computer calculates concentrations of sodium and bromine in each removed section of the oxide and the statistical error associated with the concentration, and prints this along with pertinent information.

The computer first reads in isotope half-lives, analyzer calibrations, etch-rates, and time that the calibration was done. For each wafer, it then reads in a wafer number, wafer description (i.e., the treatment done on the oxide before sectioning), background, and section area. For each of the sections removed from that wafer, it then reads in an identifying number, etching information (either an etching solution concentration plus an etch time or simply a section thickness), the time at which the count

was made, length of the count, and the number of counts respectively under the sodium photopeak and under the bromine photopeak.

The computer then takes the sodium and bromine counts and corrects them for decay, background, and interference from each other. Then, using the known analyzer calibrations and the calculated section volumes, it converts the corrected count information into sodium and bromine concentrations in each section. It then calculates the standard deviation in these concentrations due to the statistical nature of the count information.

Finally, it makes a one-page listing for each wafer. It prints wafer number, wafer description, thickness, concentration, and error information for each section from the wafer; the background, and the date on which the counts were made.

Publications

The paper $^{(2)}$ published on work done in anticipation of receiving this grant is now the prime reference on identification and distribution of Na in SiO_2 on Si.

A preliminary report of impurity diffusion and drift was presented at the IEEE-Solid-State Device Research Conference, Evanston, Illinois, June 15, 1966.

A paper entitled "Positive and Negative Ion Motion in Thermal Oxide on Silicon by Radiochemical and MOS Analysis," A. B. Kuper, C. J. Slabinski, and E. Yon, will be published in the Proc. of the Fifth Annual Symposium on Physics of Failure in Electronics, Columbus, Ohio, November 14-16, 1966.

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